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(19) (CA) **CANADIAN PATENT** (12)

(54) Separation of an Azeotropic Mixture by Distillation in a Distillation Column by a Procedure Similar to Extractive Distillation

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Abstract of the Disclosure: A mixture which is azeotropic or which behaves almost azeotropically and is difficult to separate by distillation, is separated into two pure or substantially pure fractions by distillation, by adding a further component, using a procedure which is similar to extractive distillation and is carried out in a distillation column, a section of which is divided into a feed part and a take off part by means of a separating unit which is effective in the longitudinal direction and completely or partially prevents cross-mixing of liquid streams and/or vapor streams. In this process, the azeotropic mixture is fed as bleed streams to the feed part and to the take off part, in each case at or near the top, and one of the two pure or substantially pure fractions is removed as a top product from the distillation column while the other is removed as a side product from the take off part.

Separation of an azeotropic mixture by distillation in a distillation column by a procedure similar to extractive distillation

The present invention relates to a process for separating a mixture which is azeotropic or behaves almost azeotropically and is difficult to separate by distillation, into two pure or substantially pure fractions by distillation, by adding a further component, using a procedure which is similar to extractive distillation and is carried out in a distillation column, a section of which is divided into a feed part and a take off part by means of a separating unit which is effective in the longitudinal direction and completely or partially prevents cross-mixing of liquid streams and/or vapor streams.

It is known that a two-component azeotropic mixture can be separated into its individual components by means of various distillation methods. These are essentially two-pressure distillation, azeotropic distillation and extractive distillation. These three distillation methods are described in detail by R. Billet in Distillation Industrielle, 1972, pages 223-231.

All three distillation methods have the disadvantage that two or more distillation columns are required for separating the two-component azeotropic mixture, entailing high costs in terms of apparatus and instrumentation.

It is an object of the present invention to simplify the separation by distillation of two-component azeotropic mixtures ie. to carry out the procedure in a single-stage distillation process using one distillation column.

It has been found that this object is achieved, in accordance with the invention, if the azeotropic mixture is fed as bleed streams to the feed part and to the take off part, in each case at or near the top, and one of the two pure or substantially pure fractions is removed as a top product from the distillation column while the

other is removed as a side product from the take off part.

More particularly, the present invention proposes a process for separating by distillation a mixture which is azeotropic or behaves almost azeotropically and is difficult to separate by distillation into two pure or substantially pure fractions, wherein:

a) use is made of a single distilation column having a section divided into a feed part and a take off part by means of a separating unit which is effective in the longitudinal direction and prevents cross-mixing of liquid stream, vapor stream or both of them;

b) a further component in which one of the two fractions to be separated is readily soluble, is added as an extraction agent into the mixture;

c) the mixture to be separated is fed as a bleed stream to the feed part of the distillation column, at or near the top of said feed part;

d) the fraction readily soluble in the further component added to the mixture is removed as a side product from the take-off part of the column, and

e) the other fraction sparingly soluble in the further component is removed as a top product from the column.

In accordance with a first preferred embodiment of the invention:

- the column has an undivided section located above the divided section;

- the take-off part of the divided section of the column has its top sealed off tightly from the undivided upper sections; and

- the top of said take-off part is equipped with a condenser for partial or total condensation of the readily soluble fraction of the mixture, which is removed from said take-off part.

In accordance with another preferred embodiment of

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the invention, the mixture to be separated is also fed as a bleed stream to the take-off part of the distillation column, at or near the top of said take-off part.

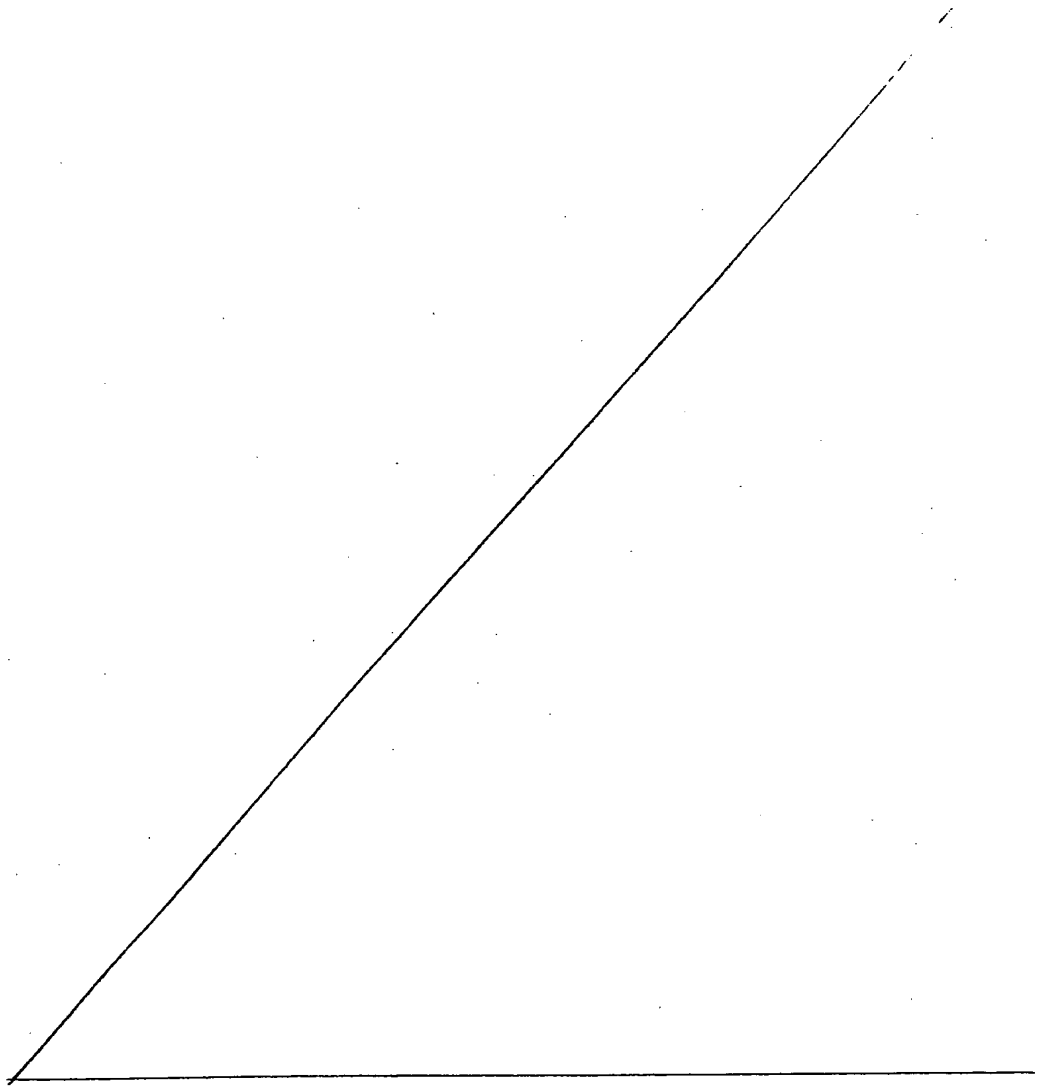
These two preferred embodiments of the invention are illustrated in the accompanying drawing, and are described in detail below.

Figure 1 shows a process flowchart for a distillation column in which the two-component azeotropic mixture is fed as bleed streams to the feed part and to the take off part, in each case at the top.

Figure 2 shows a process flowchart for a distillation column in which the two-component azeotropic mixture is fed exclusively to the feed part.

In Figure 1, a distillation column 1 (referred to below as column 1) is divided into a feed part 3 and a take off part 4 by means of a separating unit 2 which is effective in the longitudinal direction. The take off part 4 is closed at the upper end of the separating unit 2 by means of a liquid collector 5 of conventional construction, so that the liquid flowing downward from the undivided upper section 6 of column 1 is conducted completely into the feed part 3 of the column, with the result that the further component E (extraction agent) cannot pass from the undivided upper section 6 into the take off part 4. The two-component azeotropic mixture A, B is fed as bleed streams to the top of the feed part 3, which is open at the top, and the top of the take off part 4, which allows the vapor to pass through the top but prevents liquid from doing so. The bleed stream is fed in at the top of the take off part 4 in an amount corresponding to the amount of liquid required for mass transfer within the take off part 4. Because it has this function, this feed is preferably introduced in liquid form and at a very low temperature, in order to keep

the amount very small. Accordingly, in Figure 1, the two-component azeotropic mixture A, B is fed as bleed streams to the feed part and to the take off part, in each case at or near the top, while that component, A, of the said mixture which is sparingly soluble in the _____.



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further component E is distilled off via the top of the column in a conventional manner, and that component, B, of the stated mixture which is readily soluble in the further component E is removed from the take off part, in the form of vapor or liquid. As in the conventional extractive distillation, the further component E is fed into the upper, undivided section of the column, and is removed as a bottom product, either in pure form or containing small residual amounts of the readily soluble component B, and, if required, is recycled to the column.

In Figure 2, the top of the take off part 4 is sealed off tightly from the undivided upper section 6. Furthermore, the top of the take off part 4 is equipped with a condenser 7 for partial or total condensation of the readily soluble component B of the two-component mixture, which is to be removed from the take off part 4. In this case, the take off part 4 corresponds to the downstream rectification column in the conventional extractive distillation, for separating the component which is readily soluble in the further component from this component (extraction agent).

In contrast to simple distillations in columns divided lengthwise, in the novel procedure for extractive distillation the side fraction removed from the take off part passes from the feed part into the take off part only at the lower end of the longitudinal partition. Apart from the special case where the vapor rising in the take off part undergoes total condensation at the upper end of the longitudinal partition, it is even possible for flow to take place in the opposite direction, ie. some of the fraction to be removed as a side stream passes, at the upper end of the longitudinal partition, from the take off part back into the upper, common column section or into the feed part.

The decisive advantage of the novel process is that the separation of a two-component azeotropic mix-

ture into its individual components can be carried out by means of one distillation column.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A process for separating by distillation a mixture which is azeotropic or behaves almost azeotropically and is difficult to separate by distillation into two pure or substantially pure fractions, wherein:

a) use is made of a single distilation column having a section divided into a feed part and a take off part by means of a separating unit which is effective in the longitudinal direction and prevents cross-mixing of liquid stream, vapor stream or both of them;

b) a further component in which one of the two fractions to be separated is readily soluble, is added as an extraction agent into the mixture;

c) the mixture to be separated is fed as a bleed stream to the feed part of the distillation column, at or near the top of said feed part;

d) the fraction readily soluble in the further component added to the mixture is removed as a side product from the take-off part of the column, and

e) the other fraction sparingly soluble in the further component is removed as a top product from the column.

2. The process of claim 1, wherein:

- the column has an undivided section located above the divided section;

- the take-off part of the divided section of the column has its top sealed off tightly from the undivided upper sections; and

- the top of said take-off part is equipped with a condenser for partial or total condensation of the readily soluble fraction of the mixture, which is removed from said take-off part.

3. The process of claim 2, wherein:

- the further component used as an extraction agent is removed from the bottom of the column up and recycled up to the undivided upper section of said column.

4. The process of claim 1, wherein the mixture to be separated is also fed as a bleed stream to the take-off part of the distillation column, at or near the top of said take-off part.

5. The process of claim 4, wherein:

- the column has an undivided section located above the divided section;

- the take-off part of the divided section of the column has its top designed to allow the vapors to pass therethrough but to prevent the liquids from doing so; and

- the mixture fed as a bleed stream at or near the top of said take-off part is introduced in an amount corresponding to the amount of liquid required for mass transfer within said take-off part.

6. The process of claim 5, wherein:

- the mixture fed as a bleed stream at or near the top of the take-off of the column is in liquid form and at a very low temperature.

7. The process of claim 6, wherein:

- the further component used as an extraction agent is removed from the bottom of the column up and recycled up to the undivided upper section of said column.

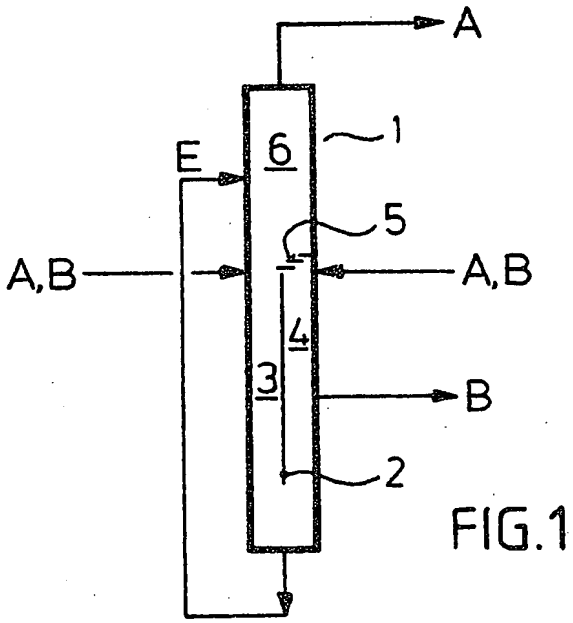

FIG.1

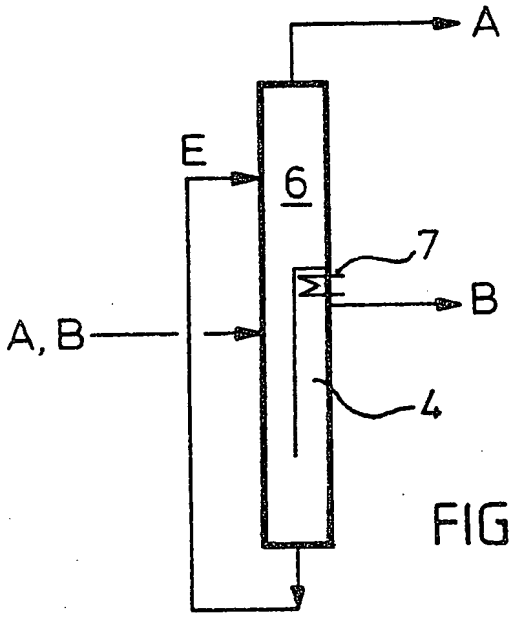

FIG. 2